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Magnetic Permeability and Relaxation Frequency in High Frequency Magnetic Materials.

M.I. Rosales, H. Montiel and R. Valenzuela
Institute for Materials Research, National University of Mexico
P.O. Box 70-360, Mexico D.F., 04510 Mexico.

ABSTRACT

An investigation of the frequency behavior of polycrystalline ferrites is presented. It is shown that the low frequency dispersion ($f <$ 10 MHz) of permeability is associated with the bulging of pinned domain walls, and has a mixed resonance-relaxation character, closer to the latter. It is also shown that there is a linear relationship between the magnetocrystalline anisotropy constant, K_1, and the relaxation frequency. The slope of this correlation depends on the grain size. Such a relationship could allow the determination of this basic parameter from polycrystalline samples.

INTRODUCTION

Many electronic devices such as switched-mode power supplies involve [1] the use of magnetic materials with high magnetic permeability ($\mu_{rel} \geq$ 1000) at high frequencies ($f \geq$ 10 MHz). The active magnetization processes in the 1 - 50 MHz frequency range have been found to be domain wall movements and spin rotation [2,3]. At frequencies higher than ~50 MHz, domain walls become unable to follow the excitation field, and only spin rotation remains. Recent studies [3,4] associate a resonance character to the permeability dispersion of domain walls. We think, however, that a more detailed investigation is needed. In this paper we present a study of the frequency behavior of polycrystalline Ni-Zn ferrites, with the aim of progressing also on the understanding of the influence of grain size on the relaxation frequency.

EXPERIMENTAL TECHNIQUES

Polycrystalline ferrite samples in the formula $Ni_xZn_{1-x}Fe_2O_4$ (with $x =$ 0.30, 0.35 and 0.40) were prepared by the ceramic method, from the reactive grade oxide reagents NiO, ZnO and Fe_2O_3. The initial wet milling of raw materials was followed by press in the shape of toroids and sintering for various combinations of time and temperature (6-96 h at 1150 °C). The furnace atmosphere was oxidizing (100% O_2 at 1 atm) in order to reduce the possibility of reduction of ferric to ferrous ions, which would reduce the electric resistivity and therefore would increase the frequency losses. Their grain size was determined by counting on scanning electron microscopy (SEM) micrographs of selected surfaces.

Real and imaginary impedances were measured in a system [2] including an HP 4192 A Impedance Analyzer controlled by a PC computer. Measurements were carried out in the 5 Hz-13 MHz frequency range at temperatures from 110 to 450 K. Real and imaginary permeability

values were calculated from impedances by using the relationship $\mu^* = KL^* = (j/\omega)Z^*$, where μ^* is the complex permeability, K is a geometrical constant (for toroids), L^* is the complex inductance, ω is the angular frequency ($\omega = 2\pi f$), j is the basis of imaginary numbers ($\sqrt{-1}$) and Z^* is the complex impedance. Note that the presence of j leads to a cross-over of values, since the real part of inductance (and therefore of real permeability) is given by the imaginary value of impedance, and conversely, the imaginary part of permeability depends on the real part of impedance. Our system allows to obtain up to 94 permeability values at discrete frequencies in less than 3 min.

EXPERIMENTAL RESULTS AND DISCUSSION

Real and imaginary permeabilities, μ' and μ'' respectively, as a function of frequency of the excitation field appear in figure 1. A clear dispersion appears, indicated in the real permeability by the decrease from a given value at low frequencies toward a lower value at higher frequencies, and by a maximum in the imaginary permeability. The complex plane, or Cole-Cole plot, where the imaginary permeability is plotted as a function of real permeability, exhibits a locus of points with the tendency to a semicircle.
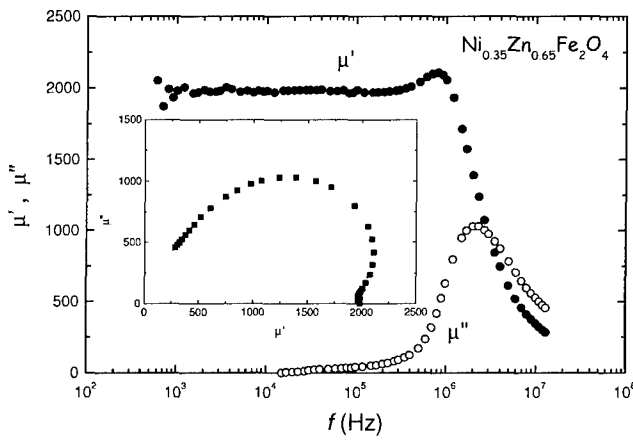

Figure 1. Spectroscopic plots of the real and imaginary parts of permeability for $x = 0.35$ ferrite. In inset, the Cole-Cole plot.

Simple dispersions typically can have either a resonant or a relaxation character. Resonant systems possess a natural frequency of vibration. If a general equation of motion is considered,

$$m \, \mathrm{d}^2x/\mathrm{d}t^2 + \beta \, \mathrm{d}x/\mathrm{d}t + \alpha \, x = F(t) \tag{1}$$

where m is the effective mass, x the displacement, β the damping coefficient, α the restoring term and $F(t)$ the excitation force, then it can be shown that a resonance dispersion occurs if the effective mass, m, is comparable to the damping term, β. The resonance frequency is then given by:

$$\omega_s = 2\pi f_s = (\alpha / m)^{1/2} \tag{2}$$

When a resonant system is submitted to an excitation of increasing frequency going through such natural frequency, the resonance appears in μ' plots as a large peak followed by a vertical drop down to the negative part of μ', finally recovering a zero value. In μ'', the resonant dispersion appears as a strong maximum. These behaviors have been observed in the case of ferromagnetic resonance in many magnetic materials, and a typical example appears in Ref. [5]. It can be shown that in complex plots, where μ'' is plotted as a function of μ' (also known as Cole-Cole plots), a resonance exhibits a full circle [6].

In a relaxation dispersion, damping is stronger than the effective mass ($m \ll \beta$) the inertia term can be neglected, and the solution of Eq. (1) simplifies to:
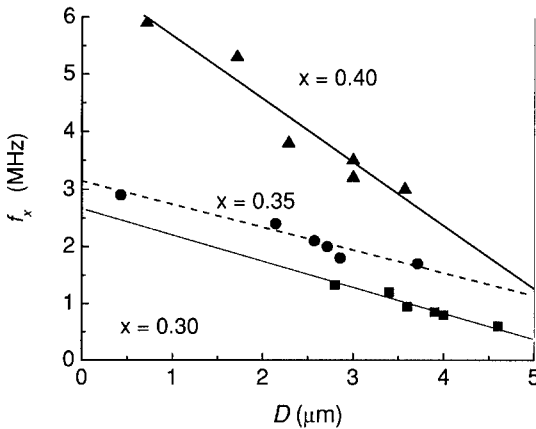
$$\omega_x = 2\pi f_x = \alpha / \beta \tag{3}$$

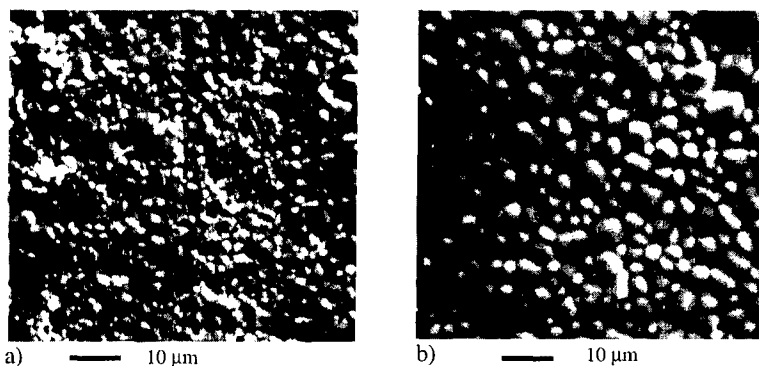**Figure 2.** Linear relationship between relaxation frequency and grain size for the studied compositions.

Figure 3. Typical microstructure of samples, for a). 6 h, b). 96 h of thermal treatment ($x = 0.35$).

In this case, the dispersion appears in μ' as a simple decreasing and in μ'' as a maximum at the relaxation frequency, ω_x ($\omega_x = 2\pi f_x$), and the Cole-Cole plot shows a semicircle. At this point, we can establish that the dispersion shown by our samples is closer to a relaxation than to a resonance, and use the relaxation frequency, f_x, in the following.

The relaxation frequency depends not only on the intrinsic parameters of ferrites, but also on the granular structure, as shown in figure 2, where a linear correlation is observed between the average grain size, D, and the f_x. Figure 3 shows the typical microstructure of samples sintered for 6 and 96 hr, in this case for composition $x = 0.35$.
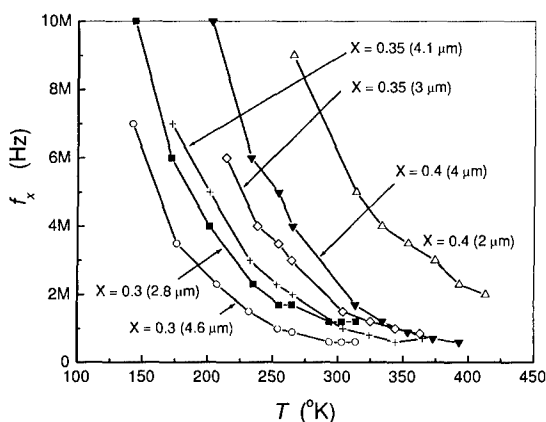

Figure 4. Thermal behavior of relaxation frequency for the studied compositions.
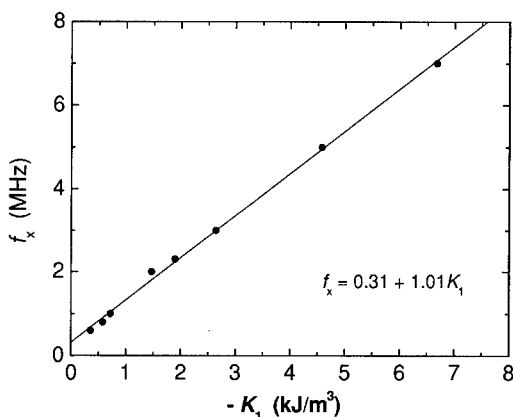
Figure 5. Relaxation frequency (f_r) as a function of anisotropy constant (K_1).

It is interesting to look at the thermal behavior of the relaxation frequency, as shown in figure 4. The decrease observed reminds in a way the decrease observed in the case of the magnetocrystalline anisotropy constant, K_1, for the case of $x = 0.35$, where single crystals were prepared [7]. When this data is plotted as a function of relaxation frequency (for each temperature), it has been shown that a linear relationship is obtained [2,8]. These results are presented in figure 5 for composition $x = 0.3$. However, the grain structure or grain size distribution has a strong influence on the relaxation frequency; a particular relationship is observed for each sample (with a different granulometry), figures 3 and 4. In order to "normalize" by average grain size, D, we have plotted $f_x D$ against the temperature, which leads to a single relationship for each composition, figure 6. In this way, the effect of grain size is taken into account.

CONCLUSIONS

We have analyzed the character of the dispersion observed in the permeability of polycrystalline ferrites and shown that it is closer to a relaxation than to a resonance. We have also shown the dependence of relaxation frequency on the grain size, as well as the effects of the latter on the relationship between magnetocrystalline anisotropy and relaxation frequency.
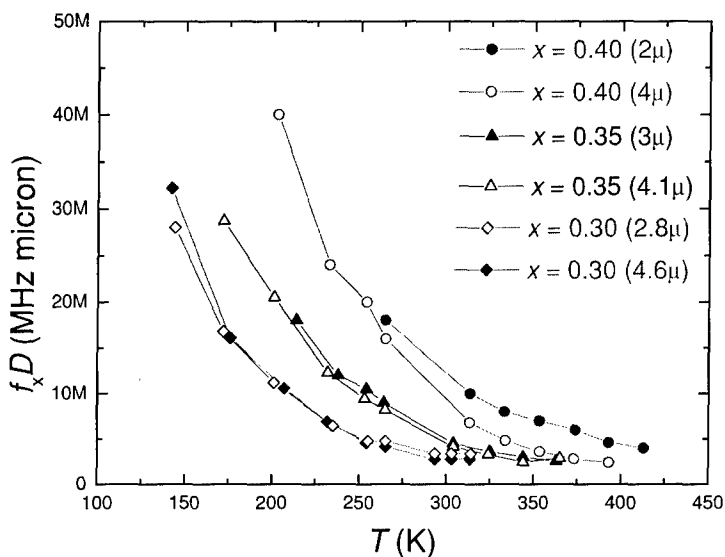
ACKNOWLEDGMENTS

Figure 6. "Normalization" of the thermal behavior of relaxation frequency by the average grain size.

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